

## Transport of ammonium perfluorooctanoate in environmental media near a fluoropolymer manufacturing facility

Katherine L. Davis<sup>a,\*</sup>, Michael D. Aucoin<sup>a</sup>, Barbara S. Larsen<sup>b</sup>,  
Mary A. Kaiser<sup>b</sup>, Andrew S. Hartten<sup>b</sup>

<sup>a</sup> URS Diamond, Barley Mill Plaza, Building 19, Wilmington, DE 19805, United States

<sup>b</sup> E. I. du Pont de Nemours & Co., Wilmington, DE 19805, United States

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### Abstract

In order to understand better the pathways for transport of ammonium perfluorooctanoate (APFO) from a point source, a focused investigation of environmental media (water and soil) near a fluoropolymer manufacturing facility (Site) was undertaken. Methods were developed and validated at  $2 \text{ Ig kg}^{-1}$  [the limit of quantitation (LOQ)] in soil, and at  $50 \text{ ng l}^{-1}$  in water. Environmental media were sampled from a public water supply well field located north of the Site, across a river. The data suggest that APFO air emissions from the Site are transported to the well field, deposited onto the soil, and then migrate downward with precipitation into the underlying aquifer.   
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### 1. Introduction

The environmental fate of perfluorinated compounds is of growing interest (Key et al., 1999; Hekster et al., 2003; Schultz et al., 2003; Stock et al., 2004; Guruge et al., 2005; Wang et al., 2005a,b; Yamada et al., 2005). Due to the unusually strong carbon–fluorine bond energy and the exceptionally high electronegativity of the fluorine atom, perfluorinated compounds often have physicochemical properties that make measuring these compounds in environmental media difficult (Kissa, 2001; Martin et al., 2004). Although a number of papers have reported measured concentrations for some of these compounds in water, biota, and air (Giesy and Kannan, 2001; Kannan et al., 2002; Yamashita et al., 2004; Barton et al., 2005; Risha et al., 2005), the transport pathways from the manufacturing facilities to the environment have not been determined. Determining transport pathways is crucial to understanding the ultimate fate of these compounds because point source

manufacturing facilities are one of the largest sources of emissions (Prevedouros et al., 2006). Recently, environmental media at and near a fluoropolymer manufacturing facility (Site) in Parkersburg, West Virginia, which uses ammonium perfluorooctanoate (APFO), were sampled for perfluorooctanoate (PFO) to evaluate potential transport pathways from the Site to groundwater, surface water and soil. Specifically, the investigation was designed to determine how PFO migrated to the well field located on the opposite side of the river from the Site.

The Site is located on an alluvial floodplain, along the southern edge of the Ohio River (Fig. 1a). Alluvial sediments underlying the Site (Fig. 1b) consist of Holocene overbank deposits (silt, clay and fine-grained sand) overlying Pleistocene-aged glacial outwash deposits (coarse-grained sand and gravel). Fractured bedrock (primarily sandy shale, sandstone, and siltstone) is located beneath the Pleistocene deposits (Simard, 1989).

The saturated alluvial deposits comprise the principal water-table aquifer used for local water supply. The Site water-table aquifer is significantly depressed due to the pumping of production wells, which induces river water

\* Corresponding author. Fax: +302 892 7621.

E-mail address: [katherine\\_l\\_davis@urscorp.com](mailto:katherine_l_davis@urscorp.com) (K.L. Davis).

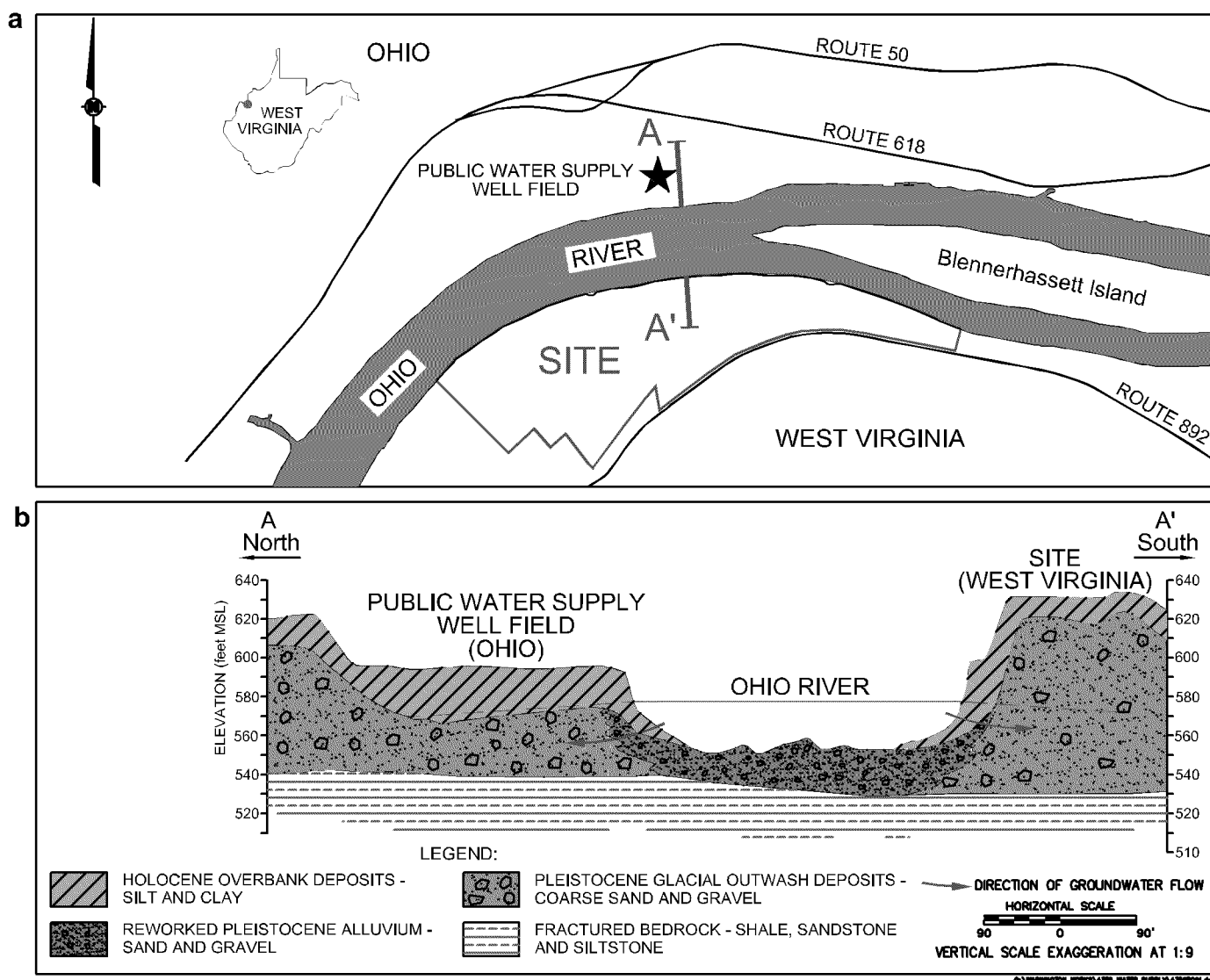


Fig. 1. (a) Site location map showing the location of the Site, the Ohio River, and the public water supply well field. Also shown is the location of cross-section A-A'. (b) Generalized cross-section A-A' showing the lithological units underlying the area and surface water recharge from the Ohio River to the well field and Site aquifers.

recharge of the water-table aquifer (Fig. 1b). A public water supply well field is located in the alluvial floodplain on the north side of the river in Ohio, across from the Site (Fig. 1a and b). The well field water-table aquifer is also depressed due to the pumping of production wells and is also recharged by river water. An investigation was conducted to evaluate potential APFO transport pathways from the Site sources to soil and groundwater at the well field because PFO was measured in groundwater from monitoring and production wells at the well field.

The well field investigation included: (1) advancing soil borings, logging soil, sampling soil and groundwater, and installing monitoring wells, (2) monitoring groundwater elevations in all production and monitoring wells, and measuring river stage, and (3) sampling groundwater from production and monitoring wells.

This paper reports the results of the well field investigation. It also discusses the results of concurrent investiga-

tions, as these studies relate to the understanding of potential transport pathways from the Site to the well field and surrounding areas. The concurrent investigations included: (1) groundwater flow and air dispersion modeling, (2) sampling of the Ohio River, and (3) surveying and sampling of private and public water supplies located within two miles of the Site.

## 2. Experimental section

The well field investigation focused on TW-4, the monitoring well that had the highest PFO concentration. (Note that PFO is the anion measured, however, concentrations are reported as APFO.) Ten temporary soil borings were advanced (Fig. 2) and soil cores were continuously logged. Soil and groundwater were sampled for PFO at various depths below ground surface. Groundwater in all wells was also sampled for PFO, and groundwater and river

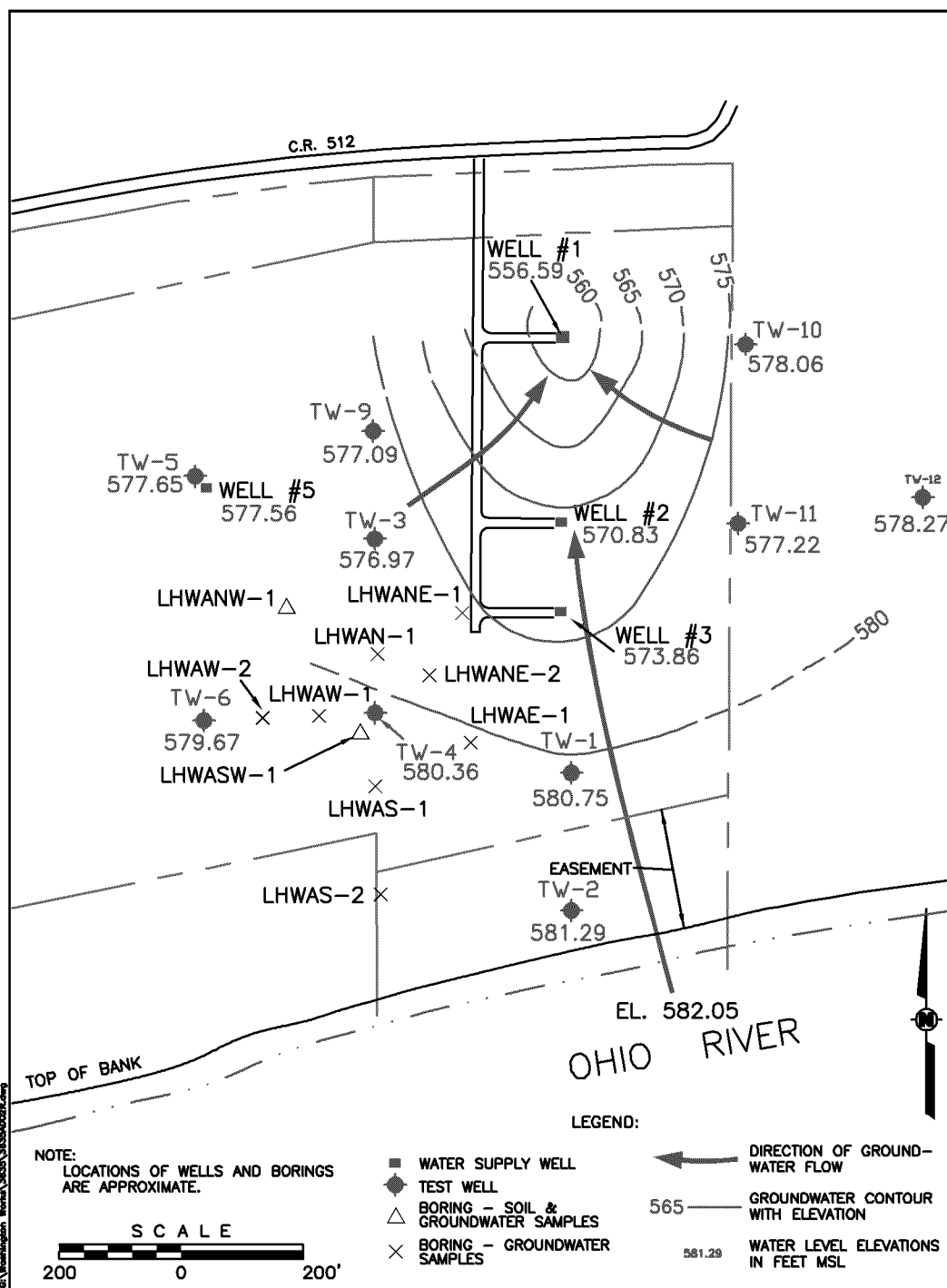


Fig. 2. Map of well field showing the location of production wells, test wells, and soil borings where groundwater, and groundwater and soil were sampled. Also shown are groundwater elevations, groundwater elevation contours and the direction of groundwater flow from the Ohio River towards the pumping production wells (data from August 2002).

water elevations were measured. Field blank samples were collected to check possible bias due to sample contamination. Field duplicates were collected as a check of sampling and analytical precision. Method (reagent) blanks, and blank and sample matrix spikes were prepared and analyzed to check bias and accuracy, respectively. Laboratory duplicates were also prepared and analyzed as a check of sample homogeneity and overall analytical precision.

### 3. Analytical methodology

#### 3.1. Materials

Perfluorooctanoic acid (PFOA) was obtained from Fluorochem USA (West Columbia, SC). [Note the perfluorooctanoate anion is measured in the liquid chromatography tandem mass spectrometry (LC/MS/MS) system.]

Methanol (HPLC grade) and sodium thiosulfate (reagent grade) were obtained from JT Baker (Phillipsburg, NJ). All water was prepared using Labconco Waterpro (Kansas City, MI) workstation (16.67 MX cm at 25 fflC) and prefiltered through a Hypercarb guard column (Keystone, State College, PA). Reagent sand was obtained from Mallinckrodt Baker (Paris, KY). Solid-phase extraction (SPE) was performed using t-C<sub>18</sub> SPE cartridges (6 cc 1 gm) obtained from Waters (Milford, MA).

### 3.2. Soil analysis

Soil samples were visually inspected for homogeneity. A representative 10 g sample was mixed with 20 ml of methanol and capped. The sample was sonicated for one hour. The solvent was decanted into a 10 ml syringe equipped with a 0.45  $\mu$ m membrane filter. A 10 ml aliquot of extract was filtered and stored refrigerated until analysis.

Calibrants were prepared from stock solutions of PFOA prepared in methanol and stored at 4 fflC. The concentrations were 1, 2, 5, 10, 20, and 50  $\text{lg l}^{-1}$ . Each calibrant was run twice using a linear calibration curve with acceptance criteria for the correlation coefficient (R) so that  $R > 0.990$ . A calibration verification standard, which is a second source of calibrant, was run with recovery of 80–120% required. The soil used for validation was taken from a floodplain located upstream that had characteristics similar to those of the well field soil. Soil spikes were made at high, medium, and low analyte concentrations, in triplicate, over a three-day period. All recoveries were between 70% and 130% and the method achieved relative standard deviation (RSD) of <20% for the spiking study, demonstrating acceptable accuracy and precision.

### 3.3. Water analysis

The C<sub>18</sub> SPE cartridges were preconditioned by first passing 10 ml of methanol followed by 5 ml of water. A 40 ml aliquot of the sample was then passed through the cartridge. The cartridge was first washed with 5 ml of 40% methanol in water and the sample was eluted with 5 ml of methanol. The final volume of the sample was adjusted to 5 ml. The LC/MS/MS method followed that of Risha et al. (2005).

## 4. Results

### 4.1. Boring logs

The stratigraphy of the well field is comprised of three lithological units (Fig. 1b). The surficial unit is a low permeability stratum ranging in thickness from 16 to 36 ft (5–11 m) that is composed of silty clay, clayey silt and sandy silt. This unit overlies a silty sand, clayey sand, and sand and gravel unit. Boring LHWANE-1, that was

completed to the underlying bedrock, showed 30 ft (9 m) of the sand unit underlain by siltstone.

### 4.2. Groundwater and river water elevation data

Groundwater and river water elevation data are presented in Fig. 2. Groundwater elevations in production and test wells ranged from 556.59 to 581.29 ft mean sea level (169.65–177.18 m mean sea level). The two Ohio River water elevations were measured at 582.05 and 582.24 ft mean sea level (177.40–177.47 m mean sea level).

### 4.3. APFO reported in soil from temporary borings

Twenty-two soil samples, including one duplicate sample, were collected from two temporary borings, LHWASW-1 and LHWANW-1. Soil APFO concentrations from the two borings ranged from non-detectable (<0.17  $\text{lg kg}^{-1}$ ; ND) to 170  $\text{lg kg}^{-1}$  in LHWASW-1 and from 6.1  $\text{lg kg}^{-1}$  to 110  $\text{lg kg}^{-1}$  in LHWANW-1 (Table 1). In LHWASW-1, the concentration of APFO was highest at the surface and decreased with sampling depth through the length of the boring. In LHWANW-1, the concentrations were also highest at the surface and decreased with depth in the boring, however, an increase in APFO was observed near the contact between the overlying silty clay and the underlying sand and gravel. In addition, the concentrations determined near the contact in LHWANW-1 were higher than those near the same contact in LHWASW-1.

### 4.4. APFO reported in groundwater from temporary monitoring wells

Eighteen samples (including one duplicate sample) were collected from the 10 temporary wells at various depths. APFO concentrations reported in the groundwater sampled from temporary borings ranged from ND (<0.01  $\text{lg l}^{-1}$ ) to 78  $\text{lg l}^{-1}$  (Table 1). Samples of the first water encountered were collected from all ten wells and were variable, covering the entire range of APFO observed. Groundwater from additional depths below ground surface was also sampled from two of the ten wells. APFO determined in these samples ranged from 0.166 to 6.22  $\text{lg l}^{-1}$ . At boring LHWASW-1, groundwater was sampled every 150 cm from the first water encountered to the bottom of the sand and gravel aquifer. APFO concentrations in this boring ranged from 0.0912 to 1.32  $\text{lg l}^{-1}$ .

### 4.5. APFO reported in groundwater from production and test wells

The range of APFO determined in production and test wells, including historical data and excluding TW-4, was

Table 1

APFO ( $\text{lg l}^{-1}$ ) concentrations reported for groundwater from various depths for the soil borings and from production and test wells, and in soil from various depths within temporary borings LHWASW-1 and LHWANW-1

Groundwater in temporary borings			Groundwater in wells	
Boring	Depth ft.	APFO ( $\text{lg l}^{-1}$ )	Well	Range of APFO ( $\text{lg l}^{-1}$ )
LHWAN1	17–22 <sup>*</sup>	50.8	Well 1	1.72–3.65
LHWANE1	21–27 <sup>*</sup>	5.58	Well 2	2.03–4.26
LHWANE1	56–58	0.662	Well 3	0.42–0.95
LHWANE2	20.7–25.7 <sup>*</sup>	1.28	Well 5	5.69–8.09
LHWAE1	21–26 <sup>*</sup>	0.416	TW-1	0.81–2.16
LHWAS1	25–30 <sup>*</sup>	ND (<0.01)	TW-2	0.081–0.103
LHWAS2	25–30 <sup>*</sup>	78	TW-3	4.17–4.48
LHWASW1	28.5–33 <sup>*</sup>	0.0912	TW-4	12.3–37.1
LHWASW1	35–40	1.32	TW-5	6.26
LHWASW1	40–45	1.02	TW-6	1.15–1.79
LHWASW1	45–50	0.376	TW-9	0.364–0.812
LHWASW1	50–55	0.166	TW-10	1.1–1.9
LHWASW1	55–56	0.254	TW-11	1.41–1.73
LHWAW1	29–34 <sup>*</sup>	ND (<0.01)	TW-12	0.758–0.824
LHWAW2	33–38 <sup>*</sup>	3.35		
LHWANW1	19–25 <sup>*</sup>	34.6		
LHWANW1	24–29	5.9		
LHWANW1	24–29 (dup)	6.22		

Soil from LHWASW-1		Soil from LHWANW-1	
Depth ft.	APFO ( $\text{lg/kg}$ )	Depth ft.	APFO ( $\text{lg/kg}$ )
0.3–0.9	170	0.1–0.5	110
5.0–5.6	13	5.0–5.5	6.1
11.2–11.7	3.4	10.0–10.5	7.5
15.5–16.0	3.3	15.0–15.5	6.9
22.1–22.6	NQ	20.0–20.5	17
26.5–27.0	ND (0.18)	21.0–21.5	18
30.0–30.5	ND (0.18)	25.0–25.5	8.4
30.5–31.0	ND (0.19)		
37.0–37.5	NQ		
37.5–38.0	ND (0.19)		
44.3–44.5	NQ		
47.5–48.0	ND (0.18)		
53.5–54.0	ND (0.18)		
53.5–54.0	ND (0.17)		
55.5–56.0	ND (0.17)		

<sup>\*</sup> First water encountered; ND = not detected at listed value; NQ = not quantifiable; LOD = limit of detection; LOQ = limit of quantification; ND < LOD < NQ < LOQ.

0.081–8.09  $\text{lg l}^{-1}$  (Table 1). The range of APFO in TW-4 was 12.3–37.1  $\text{lg l}^{-1}$ .

#### 4.6. Quality assurance and quality control

Field and laboratory blank samples were non-detect at the LOD for the target compound. Blank and matrix spike recoveries met criteria for accuracy (relative percent recovery or RPR = 70–130%) except for soil samples WWO-S-LHWASW1 (0.3–0.9), WWO-S-LHWANW1 (0.1–0.5), and WWO-S-LHWANW1 (5.0–5.5) which yielded spike recoveries ranging from 132% to 206% and may exhibit a high bias. Field and laboratory duplicate samples met criteria for precision (relative percent difference or RPD = 20%) except that laboratory duplicate precision exceeded criteria for one water sample [WWO-G-LHWASW1 (45–50), RPD = 25% and three soil samples WWO-S-LHWANW1

(15.0–15.5), WWO-S-LHWASW1 (15.5–16.0), and WWO-S-LHWASW1 (44.3–44.5), RPD ranging from 28% to 43%]. Precision above criteria may indicate samples are non-homogeneous.

## 5. Discussion

### 5.1. Boring logs

The logs for the borings were compared to logs obtained for the production and test wells. The logs show that the well field contains three lithological units, identified from ground surface downward, as Holocene overbank deposits, Pleistocene glacial outwash deposits (the well field aquifer), and Dunkard group bedrock. A generalized cross-section through the Site and the well field is provided in Fig. 1b.

## 5.2. Hydrogeology

The sand and gravel unit is the water-table aquifer utilized for local water supply and the LHWA well field has been pumped for over 30 years. The pumping of the production wells, screened at the base of the highly productive sand and gravel aquifer, results in the development of a cone of depression with groundwater flowing towards the pumping production wells (Fig. 2). Ohio River stage, 582.24 ft (177.5 m) mean sea level, is higher than the elevation of groundwater in the production and test wells, 556.59–581.29 ft mean sea level. Therefore, under pumping conditions, water from the river recharges the well field aquifer.

## 5.3. APFO transport mechanisms and migration pathways

APFO sources at the Site include air emissions and wastewater discharge, solid waste management units and single point releases (releases due to equipment failures, spills, etc.). Therefore, potential transport pathways from the Site sources to environmental media included groundwater transport, surface water transport and air emission transport and/or a combination of these three.

### 5.3.1. Potential groundwater transport pathway

A groundwater model was developed for the Site, as part of a previous investigation (DuPont Corporate Remediation Group, 1999) that confirmed that off-site migration of on-site APFO impacted groundwater was not occurring. This model was revised in 2003 to incorporate new data and to re-evaluate groundwater capture. The revised groundwater model (DuPont Corporate Remediation Group and URS Diamond, 2003a) suggests that on-site groundwater is contained by the capture zone created by the on-site pumping of the production wells, while a limited amount of off-site flow may be occurring in the far western end of the Site due to the capture zone created by the pumping of the production wells on the industrial property that is adjacent to the Site. Under current pumping conditions of production wells at the Site, at the adjacent industrial property, and at the well field, a groundwater divide exists in the Ohio River with river water flowing north on the north side of the river, towards the well field, and river water flowing south on the south side of the river towards the Site. The model predicts that the well field capture zone would only extend south across the river to the Site if all of the Site's production wells were not pumping for an extended time frame (i.e., months), a situation that has never happened. Therefore, transport of APFO impacted groundwater from the Site, through the Ohio River and to the well field is highly unlikely. In addition, the model predicts that under the current pumping conditions, no potential groundwater transport pathway exists from the Site beneath the Ohio River to the well field within the fractured bedrock that underlies the Site, the river and the well field.

### 5.3.2. Potential surface water transport pathway

The Site directly discharges APFO to the Ohio River through permitted outfalls. It is unlikely that surface water transport of APFO from the Site outfalls to the well field is a transport pathway of APFO from the Site to the well field for several reasons. The Ohio River flows from east to west between the Site and the well field, and the outfalls are on the southern riverbank, downstream of the well field. As previously discussed, pumping of production wells at the Site and at the well field results in a groundwater divide in the river with water in the northern portion of the river flowing towards the well field and water in the southern portion of the river flowing towards the Site. However, to evaluate the potential surface water transport pathway from the Site to the well field and to measure PFO concentrations in the river, water from the Ohio River was sampled along the Site reach and from as far as 26 miles (42 km) upstream and 18 miles (29 km) downstream of the Site (DuPont Corporate Remediation Group and URS Diamond, 2003b).

PFO measurements were ND ( $<0.01 \text{ lg l}^{-1}$ ) and not quantifiable [ $\text{NQ} (<0.05 \text{ lg l}^{-1})$ ] in river water upstream and along the well field and Site reach. The relative difference between those concentrations (ND and NQ) and the concentrations of APFO determined in the well field (up to  $78 \text{ lg l}^{-1}$ ), support that surface water transport of APFO from the Site outfalls to the well field through the river is an unlikely pathway. In addition, the direction of river water flow, the location of the outfall (south side and downstream) with respect to the location of the well field (north side and upstream), and the groundwater divide in the river created by the pumping of the wells on each side of the river also support that surface water transport from the Site Outfall to the well field is unlikely.

### 5.3.3. Potential air emission transport pathway

Site air emissions contain APFO, although the current concentrations are greatly reduced compared to earlier levels. The well field is downwind of the Site in the direction of the prevailing wind flow. Air dispersion modeling has been performed for the Site to predict ambient air concentrations resulting from emissions in the years 2002 and 2003 (Barton et al., 2005). This paper also reported particulate air concentrations at the Site fence line ranging from 0.12 to  $0.9 \text{ lg/m}^3$ . No vapor phase perfluorooctanoate was detected above a limit of detection of approximately  $0.07 \text{ lg/m}^3$ . Off-site modeling results indicate that air concentrations are greatest to the northeast of the Site (in the dominant direction of wind flow and the well field) and show that air concentrations decrease with distance from the Site. These results support the hypothesis that measured groundwater concentrations could be a result of deposition of APFO from ambient air.

To determine the distribution of APFO in off-site groundwater and surface water and further evaluate the air emission transport pathway, a water source surveying and sampling program was implemented for areas within a zero to one mile (0–1.6 km) and within a one and two

mile (1.6–3.2 km) radius of the Site in both Ohio and West Virginia. A total of 1261 residences were ultimately surveyed and 238 water sources, including 191 wells, 22 cisterns and 25 springs, were sampled (DuPont Corporate Remediation Group and URS Diamond, 2003b).

Statistical analysis of these data was employed to demonstrate that the concentrations of APFO in wells and spring/cisterns taken within a radius of one to two miles (0–1.6 km) of the Site were less than the concentrations in wells and spring/cisterns taken within a one mile radius (0–1.6 km) of the Site. Because approximately 85% of the samples reported APFO concentrations that were above the level of quantification, an accurate estimate of the median was determined using the nonparametric Wilcoxon rank sum test.

Results of the nonparametric Wilcoxon rank sum test showed that within each side of the river (Ohio and West Virginia) and each water source, the median concentration of APFO in water samples taken within a radius of one to two miles (1.6–3.2 km) of the Site was statistically significantly less than the median concentration in samples taken within a radius of one mile (0–1.6 km) of the Site ( $p < 0.05$ ).

This finding is in agreement with the air dispersion modeling (Barton et al., 2005) that shows lower concentrations with increasing distance from the Site.

Air dispersion modeling and the results of the surveying and sampling program in West Virginia and Ohio, support air emission transport as the most likely transport pathway for APFO from the Site to migrate to the well field and the surrounding areas.

Assuming that air transport is the primary transport pathway from the Site and knowing that PFO was measured in groundwater, it was theorized that precipitation could dissolve APFO deposited on the surface and transport it to surface water bodies and/or downward into sub-surface soils and groundwater. The results of the well field investigation were then evaluated with respect to this theory, also called the APFO transport conceptual model.

#### 5.4. APFO transport conceptual model and investigation results discussion

The APFO transport conceptual model is presented in Fig. 3. APFO in the vapor and particulate phases is emitted

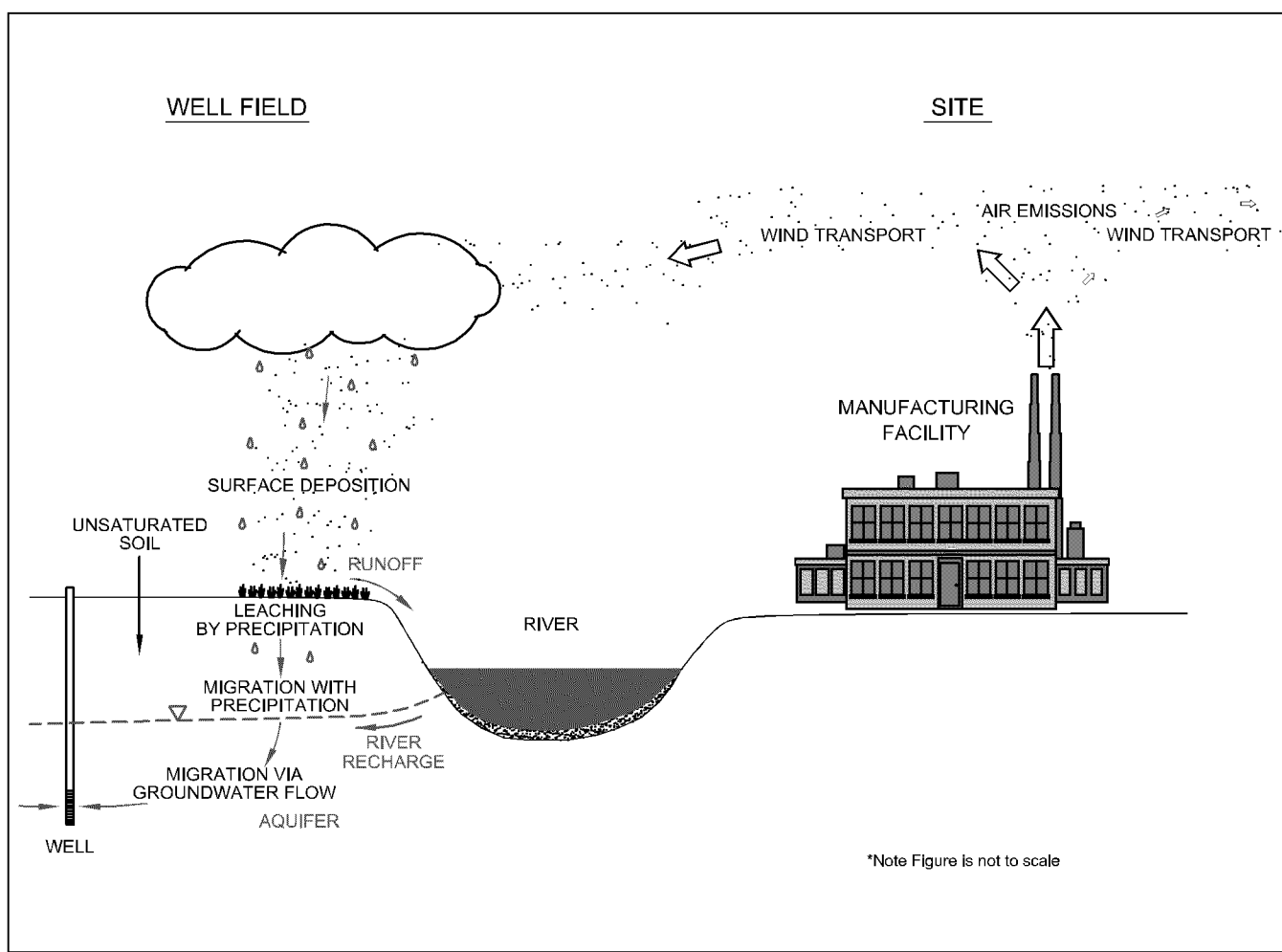


Fig. 3. Schematic of the APFO transport conceptual model. In this model, APFO from the Site is transported via air emissions by wind and is deposited on the well field surface soils. Precipitation then leaches the APFO downward through the unsaturated zone to the aquifer. Dissolved APFO, as PFO, then migrates with groundwater within the aquifer.

from the Site, transported by wind, and is deposited on the surface soil at the well field. APFO particulates are then leached by the precipitation during rainfall events from surface soils to surface water bodies and/or infiltrate into subsurface soils. These APFO particulates are then dissolved by the rainwater and migrate downward into the soil column as PFO. PFO continues migrating downward in the unsaturated zone with the infiltrating rainwater until it reaches the water-table. PFO then migrates with groundwater in the direction of groundwater flow within the aquifer.

The soil APFO data, the groundwater APFO data, and the geological data from the investigation, and the high solubility of APFO (Shinoda et al., 1972) support this transport conceptual model. The soil APFO results show that PFO is found on the surface soil and throughout the subsurface in unsaturated and saturated soil. Soil data collected from borings NW-1 and SW-1 show the highest concentrations of APFO measured were in surface soils and that lower concentrations were measured in the unsaturated zone and within the aquifer. One might expect that the concentration should be higher with increasing depth if precipitation events are constantly removing air deposited APFO from the surface and migrating it downward. However, the investigation was conducted in August, during a period of low precipitation in which APFO may have been accumulating on the surface. In addition, one might expect that the concentrations of PFO in soil would be relatively high in general, given that the Site has been emitting APFO in air for approximately 50 years. However, because APFO is highly soluble (Shinoda et al., 1972), it does not tend to precipitate or sorb to particles in the soil but it remains in solution and migrates with groundwater.

The groundwater APFO results taken during the investigation are highly variable and do not show an obvious correlation with sample depth, lithology, water level (saturated or unsaturated) or position within the well field. However, variability in the groundwater data is expected because the rate and direction of migration of precipitation (and therefore the concentration of APFO) likely changes due to several factors including the amount of precipitation migrating downward, the different permeabilities of the various lithologies encountered (silty clay versus sand and gravel), and whether the lithology is unsaturated or is saturated.

In summary, the PFO data for soil and groundwater collected during this investigation and for groundwater collected following this investigation supports the APFO transport conceptual model which describes the migration of air deposited APFO from surface soil to unsaturated subsurface soil to the aquifer.

## 6. Conclusions

The data from the groundwater flow and air dispersion modeling, the Ohio River sampling program, and the surveying and sampling program indicate that air transport is the most likely transport pathway from Site sources to

off-site environmental media and that APFO from the Site is transported via air emissions by wind and is deposited on the well field surface soils. The investigation results further support the hypothesis, or transport conceptual model, in which precipitation then leaches the APFO downward through the unsaturated zone to the aquifer. Dissolved APFO, as PFO, then migrates with groundwater within the aquifer.

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